

TGA-FTIR Standard Operating Procedures

*****You must log into FOM to use the TGA-FTIR*****

FOM and the TGA-FTIR

As with several AMSEC operated equipment, FOM is required before the instrument will connect to the computer to begin a run. This also means that if you mismanage your reservation time, FOM might kick you off in the middle of a run. This would mean the computer communication to the instrument will be cut and render that run incomplete. To avoid this, make sure to calculate your run duration times correctly and give yourself extra time to perform the run setup.

Instrument preparation

Adjust the temperature of the cell and transfer line on the TGA-FTIR interface module (below). Make sure to turn them down to 100 °C when you are done.



The maximum allowable temperature for the **transfer line is 225 °C** and the maximum temperature for the **cell is 300 °C**. You need to set the temperature as high as possible without the potential to degrade any of the products of the TGA. If the temperature is too low, you might contaminate the transfer line or cell by condensing gas into solid or liquid. This could result in long downtimes with expensive parts. The temp is modified by holding down the asterisk and using the arrows. Temp should be left at 100 °C for both heaters when not in use.

Sample preparation

Sample size requirements can vary greatly depending on multiple factors. The largest factor will be how much signal the FTIR is receiving during the run. However, you cannot use a large amount of sample, as this will give you poor temporal resolution due to the sample taking a long time to undergo the weight change.

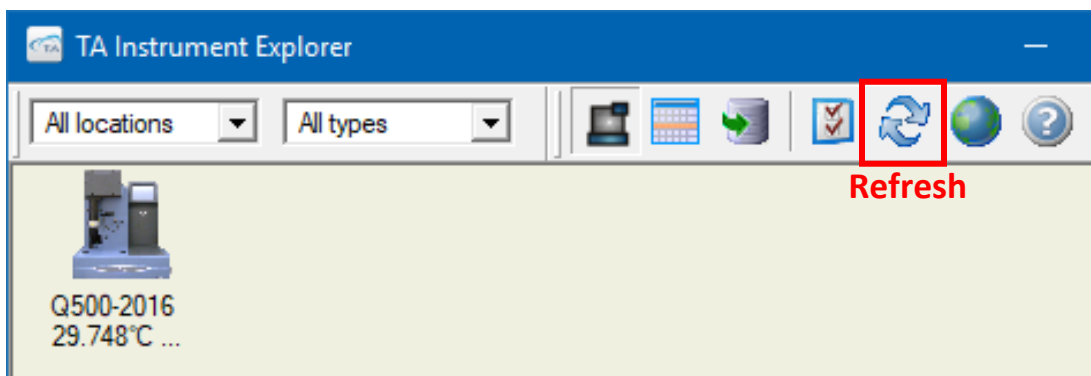
Recommended sample size is 20 – 50 mg. If your sample ends up losing less than 20% mass, you will need to use more sample. You can go up to 200 mg safely. Talk to the lab manager if you think you need to use more sample.

Liquids should not be ran in the TGA-FTIR without discussing it with the lab manager first. Liquids may pose a threat to the FTIR gas cell components.

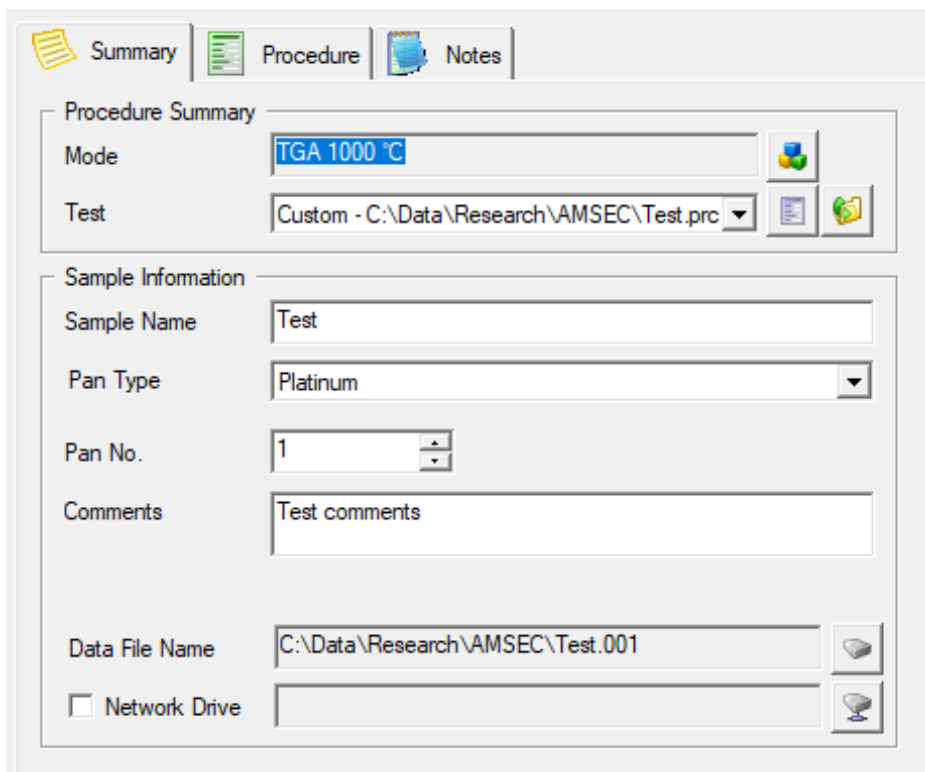
Software setup (TGA)

The TGA and FTIR software are separate programs that require a trigger from the TGA to the FTIR to sync the acquisition. Understanding this will help you set things up for successful acquisitions.

1. Open the TGA software by using TA Instrument Explorer. You may have to hit refresh several times before the Q500 shows up as available. Double click on the Q500 icon to open the software. **NOTE: You must log on to FOM to be able to connect to the instrument.**

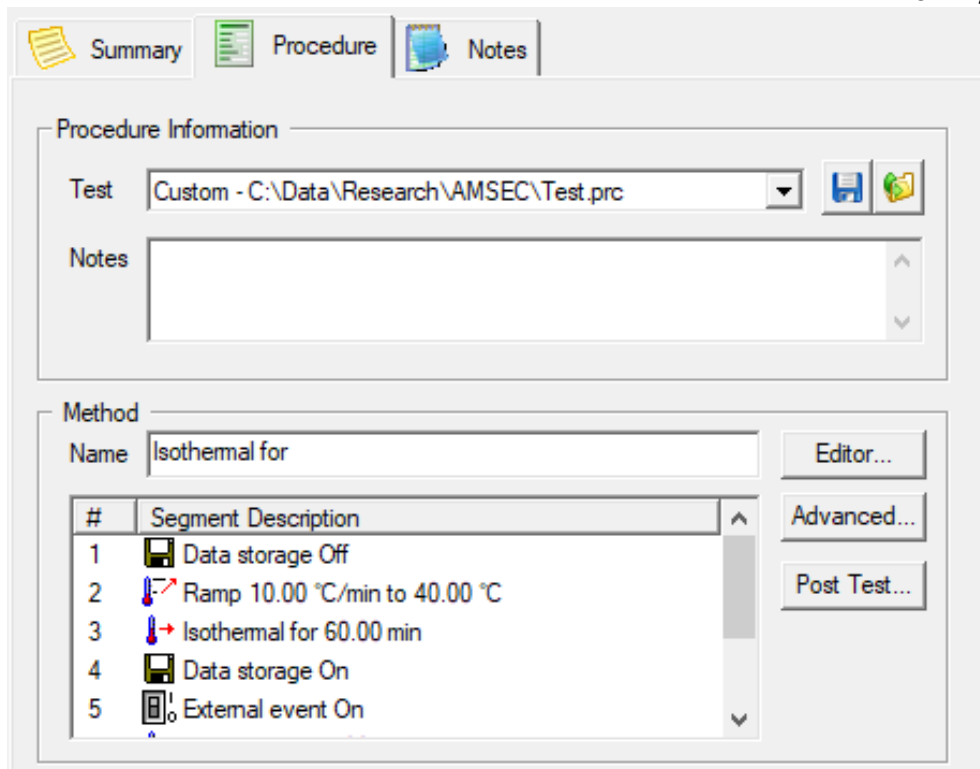


2. There should only be one run scheduled at a time. This TGA should not be set up to run multiple samples in sequence.

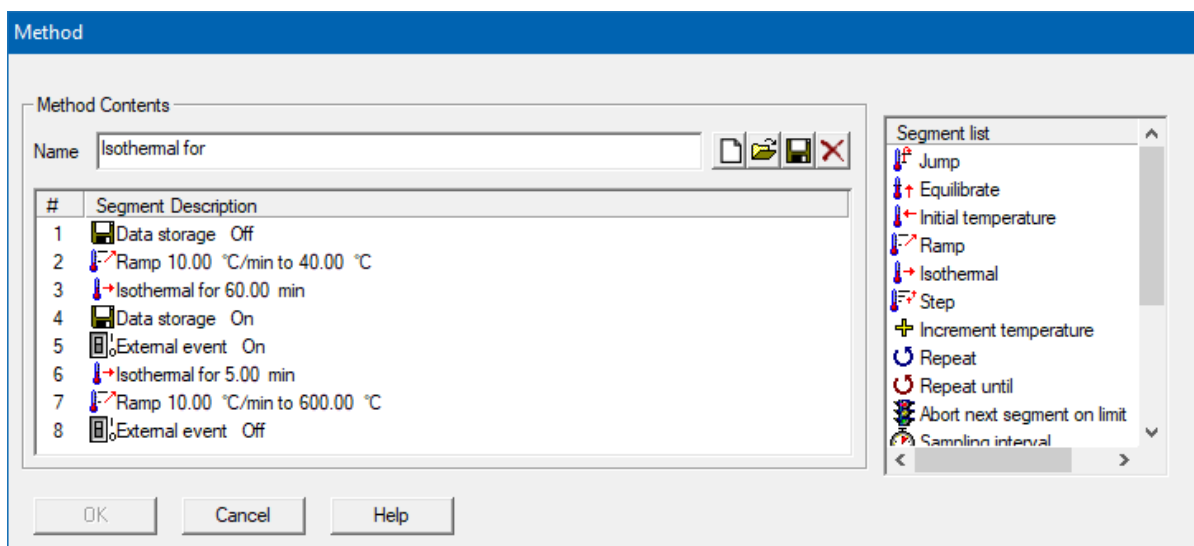


The screenshot shows the 'Summary' tab of the AMSEC software. It contains two main sections: 'Procedure Summary' and 'Sample Information'. In the 'Procedure Summary' section, the 'Mode' is set to 'TGA 1000 °C' and the 'Test' is set to 'Custom - C:\Data\Research\AMSEC\Test.prc'. In the 'Sample Information' section, the 'Sample Name' is 'Test', the 'Pan Type' is 'Platinum', the 'Pan No.' is '1', and the 'Comments' are 'Test comments'. The 'Data File Name' is 'C:\Data\Research\AMSEC\Test.001'. There is a checkbox for 'Network Drive' which is currently unchecked.

3. Name your sample and choose the proper pan type. Choose the pan position your pan is in on the autosampler and add any relevant comments.
4. Make sure you have cleaned the pan with the torch and tared it before adding your sample.
5. Choose the save location. This should be under C:\TA\Data\TGA. Please use a folder for each research group. Do not save outside of this folder as the software does not have the proper permissions for this and it will not save the data.



- The procedure tab is the most important for this technique. You need to have parameters that allow your sample to lose mass at a rate that is neither too fast nor too slow. Usually a ramp rate of 5 or 10 °C/min is adequate for this. It may take a couple runs to determine the appropriate parameters so you need to have enough sample for this as well as patience.
- You must use a custom procedure so you can program in the external event. This “External event On” step is the one that tells the FTIR software to begin acquisition.

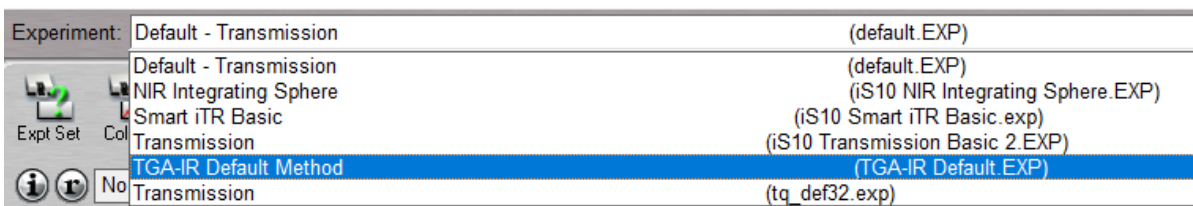


- An example of a run up to 600 °C is shown above. We wanted to have a 60-minute stabilization period for the lines to clear of air that was trapped in the TGA furnace. This is shown in line 3. This also allows water to come off. The data storage is off for the stabilization period because nothing should be happening and it clutters up the plots.

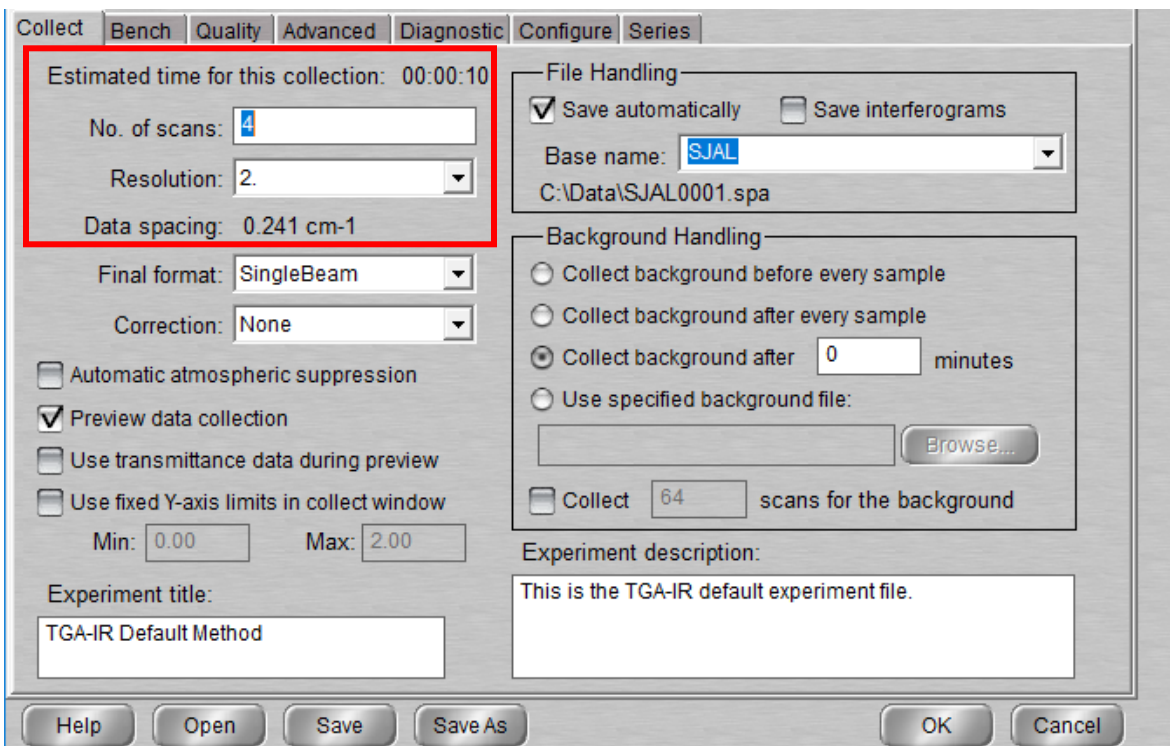
9. You can see in step 5 of the custom run segments the “External event On” option. This is the trigger that starts the collection on the FTIR. The two instruments do not communicate at all except for this one signal telling the FTIR to begin collection. The segment 8 “External even Off” does not actually do anything so in your FTIR setup, make sure you choose an adequate timeframe for your experiment so you don’t have excess and extraneous data after your run has finished.
10. The isothermal for 5 minute in step 6 will be used as the background in the FTIR, and therefore is necessary to every run.
- 11.

Software setup (FTIR)

1. Open the OMNIC software to operate the FTIR. From the experiment dropdown, choose the TGA-FTIR default experiment.

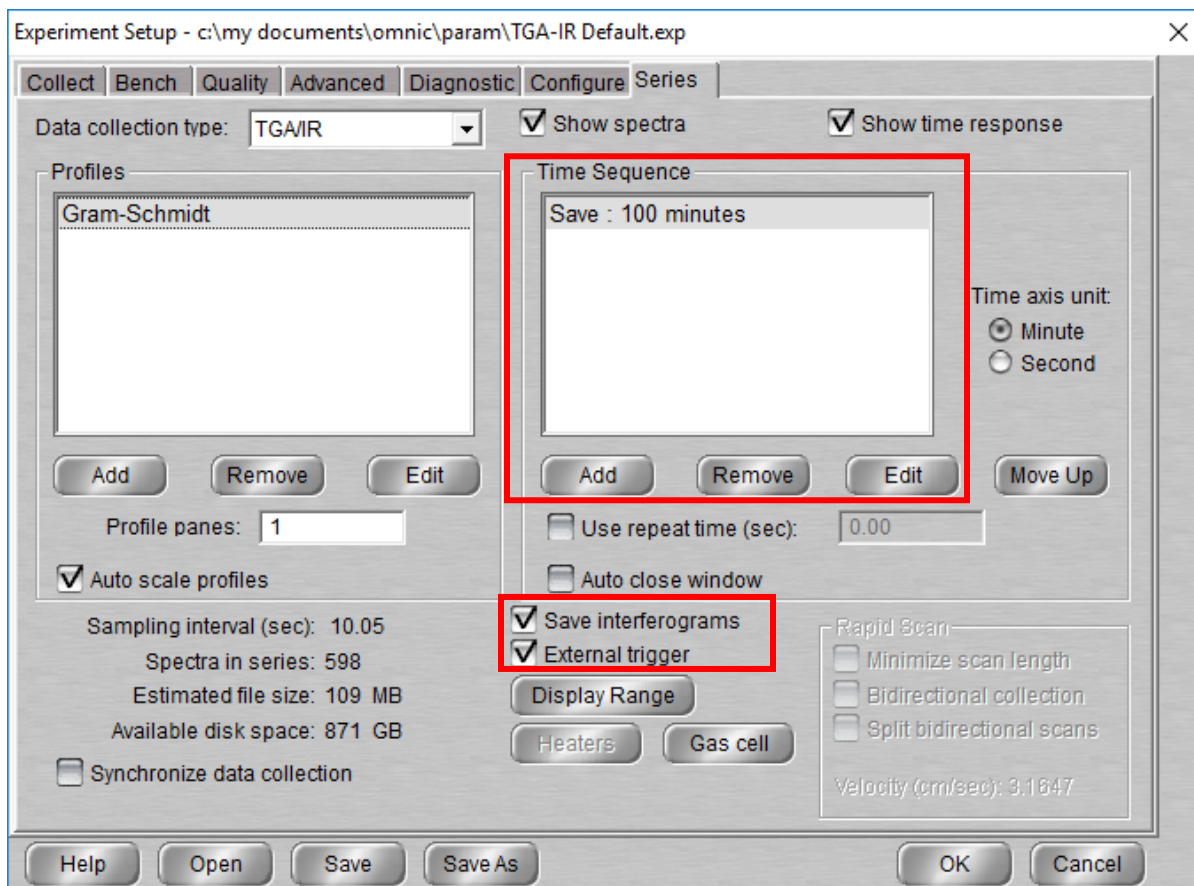


2. Under the collect menu dropdown, choose “Experiment Setup” or ctrl+e. You should see the window below. In the Collect tab, you should choose an appropriate No. of scans and resolution for your test.
3. This is based on your TGA parameters and how long your experiment is. This may need to be altered after running an experiment. For example, if you have a 120 min ramp on the TGA, we can expect the gases to come off much slower and with lower intensity. Therefore, you would want to use a higher number of scans and have each spectrum take 30 sec to 60 sec each. I recommend using a resolution of 2 or below. Higher than 2 won’t resolve many smaller peaks.
4. If you are running a faster ramp, you will likely get more signal from your sample to the FTIR and should use a lower number of scans so each scan take 5 sec to 15 sec.



NOTE: Do not change the “Final format:” of this tab from “SingleBeam” to anything else. This will be done post-experiment. If you change this, you will need to rerun the sample due to the inability to subtract a proper background.

5. Next, skip over all other tabs and go to the last one, “Series”.
6. Edit the Time Sequence by clicking on the “Save : ### minutes” and click “Edit”. Then change the time to be as long as your TGA ramp will take. Add another 10 minutes or so just to be on the safe side. For a 10 °C/min ramp from room temp to 400 °C, the save time should be ~50 minutes.



NOTE: Make sure the “External trigger” box is checked as shown above. This is the setting that tells the FTIR to use the TGA external event as the trigger to begin the collection of spectra.

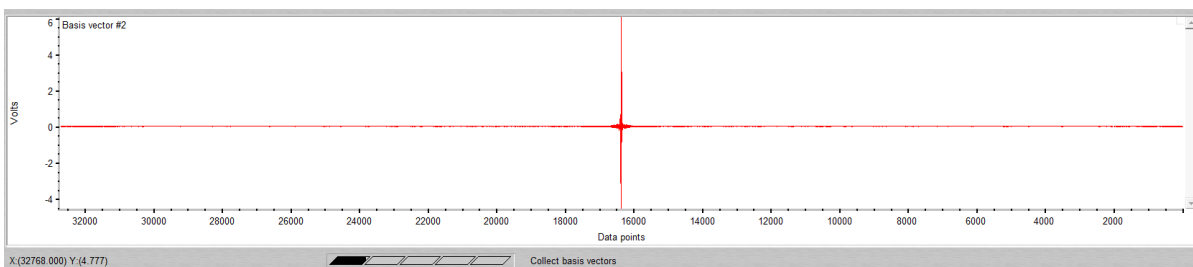
7. Click OK to accept your changes. If you dial in the settings for a run that you will be repeating several times with the same settings, you can click Save As and choose a filename to save your experiment parameters. They may show up under the experiments dropdown, or you may have to click Open from within the window above to reopen your saved parameters.

Running a sample

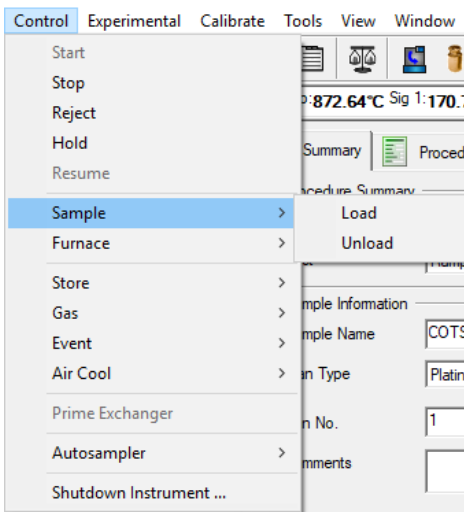
Once you have both software windows set up successfully and your sample has been loaded onto a tared pan, you are ready to begin your experiment. Start in the FTIR window by clicking “Collect Series” from the Collect dropdown menu.

Choose a save location for the files. This will produce 2-3 files, so making a folder for each experiment may be advised. If you want to save the TGA data in the same location as the FTIR data, everything will need to be saved within the C:/TA/Data folder. Click OK when prompted to collect background, this isn't the background that will be used in processing.

The FTIR will commence collecting basis vectors as seen below. This can take a few minutes, but shouldn't be a problem due to your TGA stabilization time discussed in [Step 8](#) of the TGA software setup procedure above.



In the TGA software, you can click play on the run. Getting the sample loaded and the furnace up as soon as possible will give you the best chance at purging all the air and water from the system. You can choose load sample from the control dropdown menu as seen below.



While the experiment is running, you will see each spectrum collected in the FTIR window in the bottom panel as it is collected. The TGA will show the mass percentage vs temperature. It will also show the stabilization period data, but this won't be saved in your actual data file.

Shutdown

It is very important to allow the TGA to cool naturally in order to prevent the furnace from breaking or blowing a fuse on the FTIR heaters. You must allow the TGA to cool below 200 °C before lowering the furnace. The instrument will not lower the furnace automatically after a run, you must come back to retrieve your sample after it is adequately cooled. Put the furnace back up after your sample is unloaded. The furnace must always be left up when not in use. Be sure to clean your pan when done.

You must also never use the air cool function as this will cause the FTIR heaters to blow a fuse.

Turn down the set point temps for the FTIR to 100 °C when finished.

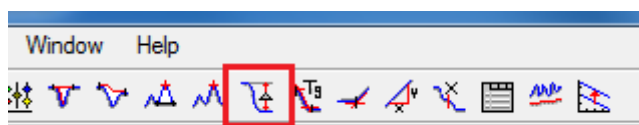
NOTE: Failure to follow the above advice could result instrument damage and major downtime.

Data analysis

There are several ways users may wish to analyze their data. Not all of the following are required to successfully view and analyze data.

Processing the thermogram

1. First open up TA Universal Analysis found on the Desktop
2. Next open up your TA file and click ok on the following pop-up box
3. You can perform many different analyses on TA data; the most important for TGA, however, is finding the weight loss for each mass loss step and finding the temperature at which those steps occur at. Begin by rescaling the window if there is unwanted data in the beginning of your experiment by right-clicking either axis and entering the domain and range values
4. To find the weight loss associated with each step, click the signal change located on the right-hand region of the toolbar. Double click the top region before any weight loss has occurred of the step of interest; then double click the bottom region of the step of interest, followed by pressing Enter. This will give you the weight loss from the first x-value to the next x-value. You can do this for as many steps as you want

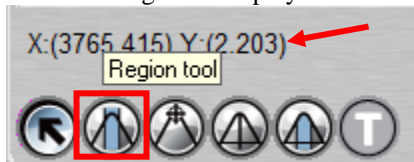


TA Universal Analysis Toolbar, where the signal change function is located (outlined in red)

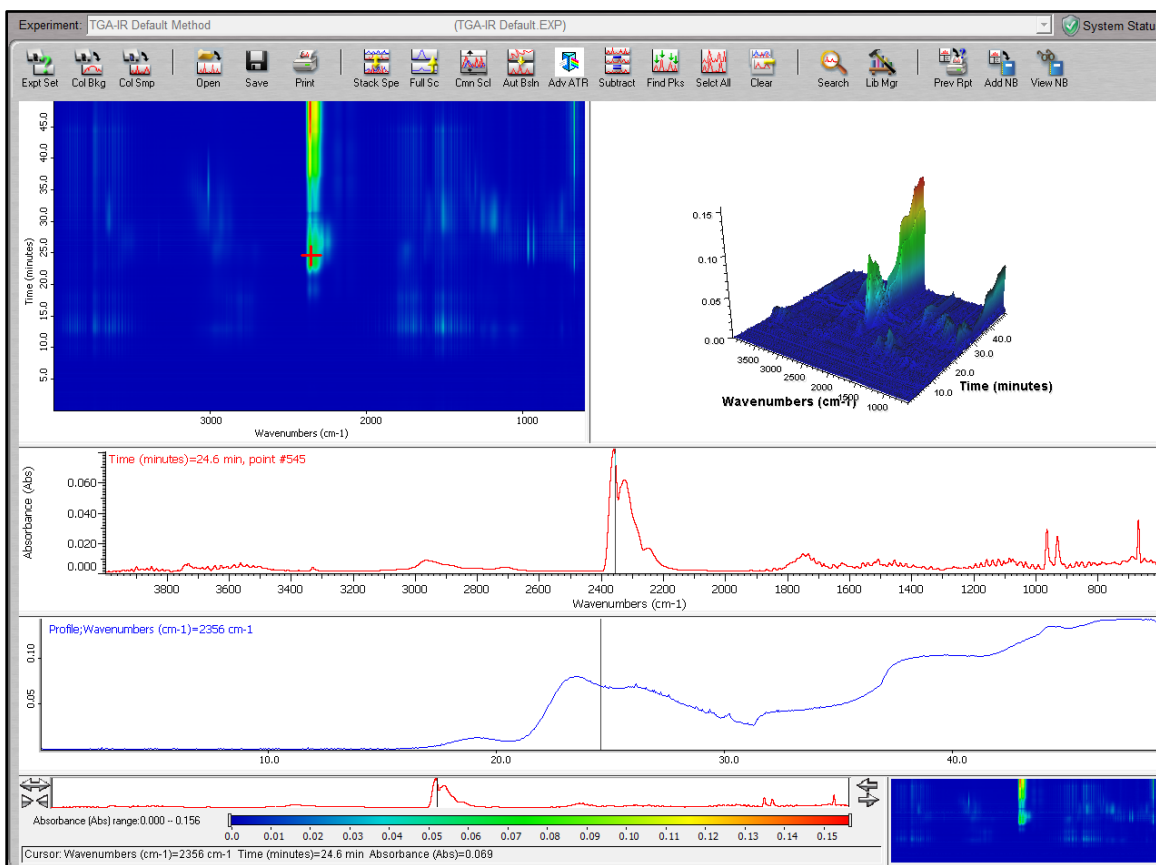
5. To find the temperature at which decomposition steps hit maximum, go to Graph→Signals, and on one of the y-axes (Y2), click Weight (%) in the signal box and Deriv. Time on the Type box and click ok. Peaks in the derivative plot are associated with individual components coming off at different times. Click the derivative plot, and click the signal maximum function located to the left of the signal change function. Click the domain where the peak is and hit Enter. This will give back the peak maximum temperature
6. To export this data go to File→Export Data File→File and Plot Signals

Processing the IR Series

1. Begin by opening up the series file on OMNIC
2. First subtracting the background is necessary.
 - a. First, select the time region in the Gram-Schmidt plot you wish to use as the background. We have it set in the instructions as a 5 minute background. Use the region tool to click and drag the time in the plot to use. The time range will display in the X: Y: section right above the region tool shown below.



- b. Then, you will coadd the selected time range to a single plot. To do this go to the series dropdown and choose "Coadd Region". The lower plot should now contain the first 5 minutes of spectra and you should see the following in the top left of the lower plot. **Coadded spectrum: 5.23 - 0.17 min.**
 - c. Right click on the lower plot and click Copy To→New Window, and click ok. Save this new spectrum with the rest of your data. Using the notation of "BKG" within the filename is common to denote the filetype.
 - d. Exit out of the background window and go back to your series window. From here click Process→Reprocess, and put the Final format to Absorbance. Click browse and select the file you just made as your background file. Click ok and the background will be subtracted from all spectra of your series file.
3. After this you can also choose Process→Automatic Baseline Correct to clean up any drifting baselines
 4. To look at the 3D plot with the contour map, click Series→Show Series 3D Window. From this window you can track how certain peaks grow in and out as time goes on, and you can also look at the linked spectrum at whatever point your cursor is at



Series 3D Window of a TGA-FTIR run, showing the contour plot with color representing the intensity, a 3D plot, the linked spectrum, and a tracer plot at a specific wavenumber

5. You can copy out each individual spectrum of interest by clicking on the heat map. The spectrum will show up down below and you can right click and choose copy to new window where you can then save the data and process it.

Data extraction

To save each individual spectrum as a .csv the following is required.

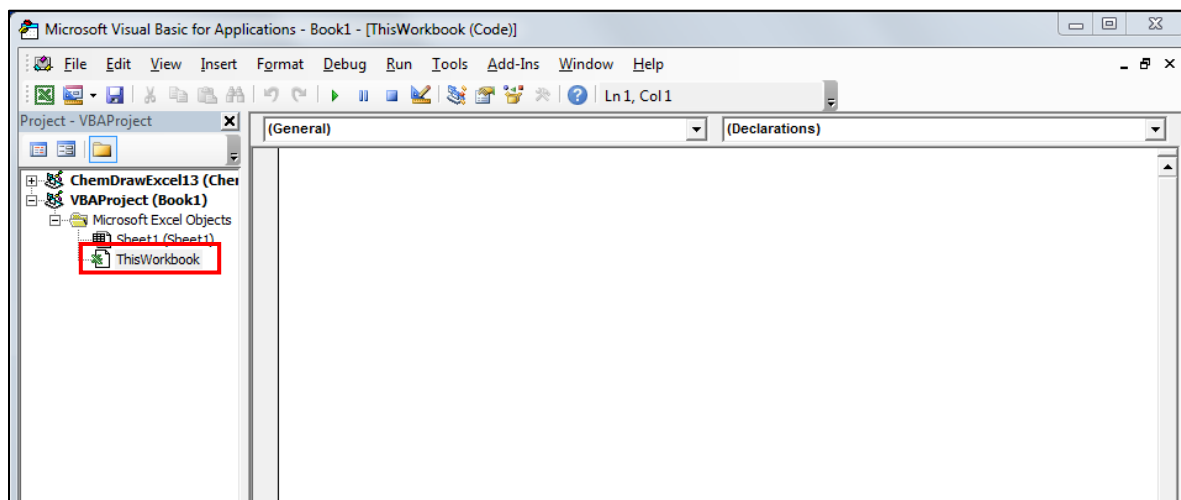
1. Under the Series dropdown choose "Split Series File", click "Set Path" to select the location for your files. Set a base name all your files will share and click OK.
2. Once complete, open all of these OMNIC .spa files in one window by shift-clicking all the files and clicking open. Depending on how many files there are it may take a while



3. Afterwards **Select All** spectra in the window (they should all be highlighted red) and go to File→Save As, select the file format as .csv, and click ok. From here the save box will pop up for every file, prompting the user to hold the enter button down until all files are saved as .csv. This can easily be accomplished by placing something on the enter button until completed.

Compiling Individual .csv files into 1 XYZ Plot

1. Open a blank spreadsheet in Excel, and press Alt+F11, opening up VBA.



Blank spreadsheet in Excel with VBA (right) and ThisWorkbook highlighted with the red box

From here click ThisWorkbook on the left hand side Project Toolbar. In the code box paste in the following:

```
Sub simpleXlsMerger()
```

```
Dim bookList As Workbook
```

```
Dim mergeObj As Object, dirObj As Object, filesObj As Object, everyObj As Object
```

```
Application.ScreenUpdating = False
```

```
Set mergeObj = CreateObject("Scripting.FileSystemObject")
```

```
'change folder path of excel files here
```

```
Set dirObj = mergeObj.GetFolder("E:\3D plot")
```

```
Set filesObj = dirObj.Files
```

```
For Each everyObj In filesObj
```

```
Set bookList = Workbooks.Open(everyObj)
```

```
'change "A2" with cell reference of start point for every files here
```

```
'for example "B3:IV" to merge all files start from columns B and rows 3
```

```
'If you're files using more than IV column, change it to the latest column
```

```
'Also change "A" column on "A65536" to the same column as start point
```

```
Range("A2:IV" & Range("A65536").End(xlUp).Row).Copy
```

ThisWorkbook.Worksheets(1).Activate

'Do not change the following column. It's not the same column as above

Range("A65536").End(xlUp).Offset(1, 0).PasteSpecial

Application.CutCopyMode = False

bookList.Close

Next

End Sub

1. Change the range accordingly in the code above, based on how many files you have and how many rows are in each one (e.g. 78 files, each containing 4,000 rows → 4,000 x 78 = 312,000 for the range value)
2. Designate the folder from which the files are located in the code above
3. Click play on the VBA toolbar
4. Once complete, go to the excel spreadsheet and create a temperature column in the C column using the following code for all entries:

=INDIRECT("D"&INT(ROW(C3736)/3736+1))

5. If each file contains more/less rows than 3736, change the code above accordingly. The amount of rows must match for the coding above to work properly
6. In the D column create the temperature range and appropriate step rate depending on your experimental conditions and how many files you're including in creating your 3D plot
7. Once done, copy and paste the wavenumber, absorbance, and temperature columns and paste into Origin

Plotting the 3D IR

1. Designate the X, Y, and Z columns in the Origin spreadsheet and go to Plot→3D Surface→Color Map Surface. Afterwards make sure to change the direction of the wavenumber scale to normal format. Label the axes, change color scheme, change gridlines, change mesh, etc.

Instrument Hazards and Best Practices: TGA-FTIR

This document will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety-training document that each user has attested to have reviewed at fom.wvu.edu/documents

Lab coats are to be provided by the user unless special hazards exist in which case they are located at the PPE station.

Hazards:

- Chemical exposure
- Infrared laser radiation
- Extreme Temperatures (Up to 1000 C)
- Hot Flames (propane torch)

1. Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-155-215).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

2. Infrared Laser Radiation

Use of controls or adjustments or performance of procedures other than those specified in your printed and electronic documentation may result in hazardous radiation exposure. Do not alter or attempt to remove the laser head from its protective housing or attempt to remove the shields that surround the laser head. Exposure to laser energy and high voltage may result.

Never stare directly into the laser beam or at its bright reflection. Avoid skin contact with the laser beam or its bright reflection. Never tamper with the laser head. Exposure to laser light or high voltage may result in injury.

3. Extreme Temperatures

The TGA operates from room temperature up to 1000 C. When the furnace is in the operating position of up, it is very safe and not harmful to the touch. When the furnace is brought down after a run completes, it will be very hot. Avoid contact with the furnace during this period of cooling after a run. Also avoid coming into contact with the cooling air stream exiting the top of the furnace as this may be hot as well. If no air cool executed after a run, it may remain hot for up to an hour. When an air cool is used after a run, it should be cool after 10 to 15 minutes. As a common practice, users should never touch the furnace. This is due to not only the temperature risks, but to keep the furnace from becoming contaminated.

The transfer line and TGA accessory of the FT-IR are heated to temperatures up to 300 C. Precautions should be taken to avoid contact with these apparatus. These apparatus should not be allowed to contact anything that may be affected by such temperatures. Severe burns may result if these precautions are not taken.

4. Hot Flames

The procedure for cleaning the TGA pans includes the use of a propane torch to heat the samples pans up. The torch flame reaches temperatures over 1200 C and will rapidly heat or set aflame anything it comes in contact with.

Standard laboratory clothing is required, including a lab coat. A minimum of impact glasses are required for this procedure unless liquid chemicals are present. It is recommended to wear flame resistant gloves and a flame resistant lab coat that are provided in the PPE station. Please put them back after use.

Torch operation is straightforward. Avoid using the torch anywhere except near the TGA as care has been taken to prevent anything flammable or combustible from being located in that vicinity. If you notice anything flammable or combustible near the TGA, please contact the lab manager immediately. To use the torch, turn the brass knob on the back counterclockwise until it stops. Then turn the yellow knob on the front to the on position. Then pull the yellow knob and hold it down. If the torch does not ignite at first click, try again until it does. Avoid pointing the torch in the direction of any persons or equipment or furnishings. Use the torch at least two feet from anything.

Standard practice should be to use the long tweezers provided near the instrument to hold the pans during torching. The tweezers should be over 8" and made from steel to prevent rapid heat transfer and temperature resistance. If these tweezers cannot be located, do not perform this procedure until they have been acquired. Contact the lab manager for assistance. To torch the pans, grab them with the long tweezers. Keep your hand being used to grip the tweezers as far from the flame source as possible. Grab the pan with the long tweezers by the corner of the wire. Avoid grabbing it by the top of the wire. Use the propane torch to heat the sample pan to red hot for at least 5 seconds or until no further change is observed. Let the pan cool down for 3 seconds before setting down. **Caution:** the tweezers will remain hot for several minutes after torching. Take care to avoid coming into contact with the tweezers or letting the tweezers encounter anything they may damage due to high temperatures.